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Research Article

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Synthesis and characterization of commercial pure titanium-nickel alloy behavior reinforced with titanium diboride

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Abstract

Commercial pure titanium alloy with Ni-TiB₂ ceramic additions (5, 10, 15 and 20 vol.%) were synthesized through the spark plasma sintering approach with sintering temperature of 1000 °C, the heating rate of 100 °C/min, holding time of 5 min at a constant pressure of 50 MPa. The study investigated the effect of Ni-TiB₂ on the densification, phase change, microhardness, microstructure, and wear properties of the sintered titanium-based composites. Results showed that Ti-Ni-TiB₂ composites relative density ranges from 97 to 99 %, while microhardness values increase with addition of nickel and titanium diboride from 228 to 587 HV_{0.1}. The microstructural evolution shows that pure titanium transformed from lamellar phase to equiaxed alpha phase upon addition of nickel alloy and further get refined with a distinct grain boundary comprises of titanium diboride around the boundaries. The average coefficient of friction for the titanium-based composite was higher for commercially pure titanium (0.73) while the addition of TiB₂ exhibit (0.66, 0.63, 0.58, 0.55 and 0.46 respectively) improvement in the wear behavior.

Keywords: Titanium; nickel; TiB₂; dispersion; microstructure; wear

1. Introduction

Titanium and titanium alloys have been utilized in different engineering applications because of their attractive properties, for example, low density, high specific strength, biocompatibility and exceptional corrosion resistance [1,2]. Despite the properties that titanium alloys with usage in various engineering applications, they are restrained for high-temperature applications usage due to their poor tribological properties. The way toward improving the mechanical properties of titanium alloys can be accomplished through the addition of various alloying elements, for example, alpha stabilizer, neutral, beta isomorph and eutectoid stabilizers. Nickel is beta eutectoid stabilizers which at high temperature, nickel and titanium alloy form intermetallic compounds which is ductile with better plasticity, high impact resistance and good damping properties [3–5].

Titanium diboride (TiB_2) have attracted increasing consideration from various researchers because of its various applications such as, cutting tools, wear applications, high-temperature structural materials and lightweight impact resistance protective layer material, because of its exceptional mechanical properties, for example, high melting point, high hardness, high Young's modulus, good abrasion resistance, high strength, thermal conductivity and chemical stability [6–8].

Metal matrix composites have shown remarkable potential for the structure and improvement of innovative new advanced materials which is achieved in consolidating ceramic as methods for strengthening metal matrix to improve its mechanical properties [9]. Metal and its alloy don't completely possess some degree of properties required in essential materials and this driven the improvement through metal matrix composites by reinforcing it either utilizing particulate or ceramic into the metal matrix [10,11].

Spark plasma sintering is another sintering method that permits sintering of powders at lower sintering temperatures, rapid heating, holding and cooling time when identified with different conventional sintering technique [12]. In SPS process, because of its highlights, such as, raw powders and graphite die are directly heated with a pulsed current which prompts the making of sparks between the powder particles as a result generate spark discharge and mass transfer which can be accomplished rapidly and further simplicity expulsion of oxide contaminants from the surface of the particles [13]. The application of pressure can deform the particles and accordingly permitting the production of stronger connections between neighboring particles whereby the

electrical field can help in atom diffusion [14]. Spark plasma sintering methods have demonstrated that rapid time and heating rates consolidation has been advantageous in preventing grain development and control of microstructures thereby retaining the grain size and achieving improvement in mechanical properties [15,16] with high relative densities in a short timeframe.

In this study, TiB₂ particle-reinforced titanium-nickel based composites were successfully created utilizing a spark plasma sintering method. The effects of Ni with ceramic particle (TiB₂) additions at the distinctive volume per cent composition on the densification, hardness, microstructures and wear behaviour were examined.

2. Experimental procedure

Titanium, nickel and titanium diboride powders were milled using a planetary ball mill (Retch 400 PM) with alumina cup and balls for 8 h at an interval of 10 min, powder to ball ratio of 10:1 and rotation speed of 150 rpm were used during the milling process. These parameters were selected to avoid powders from been heated up to prevent cold welding of the powders and avoid unwanted phase formation that could occurs within the powders during milling operation. The admixed powders were solidified utilizing spark plasma sintering machine (HHPD-25 FCT Germany) and further compacted in graphite die with inner distance across of 20 mm and 5 mm thickness at a steady temperature of 1000 °C, heating rate of 100 °C/min, applied pressure of 50 MPa and holding time of 5 mins under a vacuum atmosphere.

The density of the sintered samples was estimated utilizing using Archimedes technique as per ASTM B962-15 standard. The titanium matrix composites were precisely weighed both in air and de-ionized water utilizing an electronic measuring balance. Microhardness Vickers machine (FALCON 500 series) was utilized to do the hardness test at a full load of 300 gf with a dwell time of 15 s. The phase identification of the sintered composites was performed utilizing XRD PW1710 Philips diffraction with Cu target K α radiation at 40 kV and 40 mA. While X'pert Software was utilized to identify every constituent phases of the sintered composites. The polished samples were etched utilizing Kroll's reagent and further examined utilizing scanning electronic microscope (Vega/Tescan model) equipped with an energy dispersive range (EDS) analyzer with an accelerating voltage of 15 kV.

The wear test was completed utilizing a tribometer pin-on-disk friction module procedure. A stainless-steel ball with 6 mm in diameter was utilized as a counter face rubbing against the titanium-based composite. A load of 10 N was applied with a rotational speed of 300 rpm under a dry rotary condition at ambient temperature. The mean coefficient of friction (COF) was determined and reported for all materials. The volume loss (V) of the titanium-based composites was determined utilizing the below equation 1:

$$V = \frac{\Delta w}{\rho} \quad (1)$$

Where Δw = weight before test-weight after test and ρ = density of the titanium-based composites. The wear rate (W) was then determined to utilize the below equation 2:

$$W = \frac{\Delta w}{L \cdot \rho \cdot F} \quad (2)$$

Where Δw is weight loss difference (before-after tests), ρ is density of the titanium-based composites, L is sliding distance and F is the applied force.

3. Results and discussion

3.1 Characterization of starting and admixed powders

Figure 1a-c shows the SEM morphology of titanium, nickel, titanium diboride and admixed powders. Figure 1a shows titanium alloy powder is spherical and non-porous with a few agglomerated satellites adhering to the bigger particles while titanium diboride is little irregular particles. Figure 1d-e revealed the micrograph of the composite containing 5 vol.% Ni and 20 vol.% TiB₂. The powders resulted in uniform relatively dispersion within the surface of the titanium alloy particles. Also, the shape of the titanium particles can be found to have change a little with some agglomerated particles which might be due to cold welding during the mixing operation. The process of powder blending approach is typically noticeable by the homogeneity of the blended powder [17]. Figure 1f revealed the EDX of the element present after the milling process. After mechanical alloying, the grain size of all powders is reduced, and the titanium particles are protected by fine grains of Ni and TiB₂ particles. The decrease of grain size, homogenous dispersion of particles because of changes during processing shorten the dispersion way and increasing defects density are significant points of interest of mechanical alloying [18].

Fig. 1. SEM morphology of the powders; (a) Commercial pure titanium, (b) Nickel, (c) Titanium diboride, (d-e) admixed powders of Ti-Ni with 5 and 20 vol.% TiB_2 and (f) EDX of the admixed powders.

The XRD spectra of the blended powders with different amount of titanium diboride have shown in Fig. 2 with controlled peak intensity. The blended powders present some similar patterns and some new phases formation after mixing of the powders while remained the α phase. It was observed that the α phase is characterized by the (100) reflections at a peak position of $2\theta=34.27$ and the diffraction peak was slightly decrease upon the increase of the reinforcement in the whole composite material.

Fig. 2. XRD patterns of the admixed composite powders

Figure 3a-c revealed the consequence of sintering temperature, displacement and shrinkage rate as a function of time for the sintered composite material. The sintering temperature of 250 °C was kept for few minutes and then raise up to 1000 °C and held for 5 min and further cooled down to room temperature. Figure 3b display an increase in displacement as the temperature raises which is because of rearrangement of the powders with the effect of pressure exerted upon it that causes discharge of trapped air and gas within the compact. The sintering processes goes together with neck growth due to the collective effects of evaporation and condensation which rises sintering temperature [19]. Thus, an increase in displacement behaviour shows that transformation took place in the physical state and structure of the powders. Figure 3c demonstrate the shrinkage rate that occur during the total sintering time of the spark plasma sintering process. The shrinkage rate display two distinct peaks. Stage 1 shows a combination of early rearrangement of the powders, gas removal, formation of spark presence and the shrinkage rate arose with an increase in temperature causing bonding between the powder particles which resulted in melting, local deformation of particles arrangement, growth of the sintering necks at contact zone and plastic flow movement [20]. At the second stage, which signifies a little deforming zone because of the short holding time of 5 min while the sintering operation ends with character of decrease in shrinkage rate.

Fig. 3. Sintering parameters during SPS process against time: (a) sintering temperature, (b) relative displacement and (c) shrinkage rate.

3.3 Density and hardness measurement of sintered titanium-nickel based composite

Figure 4a shows the effect of sintering parameters on the densification behavior. The relative density decreased with the addition of reinforcement from 99 to 97 %. However, the incorporation of ceramic reinforcement into materials tends to decrease their density [21]. The decrease shows that some few porosities might be present in the composite material. Figure 4b shows the microhardness value which increases as the reinforcement is addition in different percentage. The increase in microhardness can be attributed to the high hardness characteristics of the additional ceramic additives which prevent dislocation movement and give a good resistance to plastic deformation of the titanium matrix composites [22]. Also, the increase in hardness of the titanium matrix composite can be attributed to the homogenous dispersion of the particles.

Fig. 4(a) Relative density and (b) Microhardness values of the sintered titanium-based composite.

3.4 Microstructural characteristics of the titanium-nickel based composite

Figure 5 shows the SEM micrograph of the produced titanium-based composites (Ti reinforced with 5% Ni and 5-20 vol.% TiB₂). Dispersion of the reinforcement should be detected in the titanium matrix and to prove that spark plasma sintering technology is actual to process these materials. This technique allows densification of the matrix with effective dispersion. However, some agglomerated particles should still be identified which might be due to high specific surface area of the particles which causes clustering. Figure 5b-f reveal that the microstructural evolution of pure titanium transformed from lamellar phase to equiaxed alpha phase upon addition of nickel alloy and further get refined with a distinct grain boundary comprises of titanium diboride around the boundaries.

Fig. 5. SEM microstructure of sintered composites; (a) Ti alloy, (b) Ti-5 vol.% Ni, (c) Ti-5 vol.% Ni-TiB₂, (d) Ti-10 vol.% TiB₂, (e) Ti-15 vol.% TiB₂ and (f) Ti-5 vol.% Ni-20 vol.% TiB₂.

3.5 X-ray diffraction of titanium-nickel based composite

Figure 6 shows the XRD patterns of commercially pure titanium alloy and varying volume fraction of TiB₂ with nickel carried out at a sintering temperature of 1000 °C and the respective miller

indices. Addition of nickel into titanium alloy causes a reduction in the intensities peak, with a slight shift in the peaks which, could be as a result of high tensile stress of α phase [23]. However, diffraction peaks of the sintered titanium-based composite confirm the presence of both α and β peak, which are predominantly phases [24]. On the other hand, an increase in the peak height with the addition of 5-20 vol.% TiB_2 was observed. Generally, the decrease in the peak height of the titanium-based composite could likewise be credited to the x-ray absorption effect [24]. Thus, some peaks were observed which are different from the primary titanium alloy such as (106), (303), (200) and (112) at $2\theta=53.28^\circ$, 63.49° , 76.17° and 77.62° respectively.

Fig. 6. XRD patterns of sintered titanium-based composite: (a) Titanium alloy and (b) varying volume fraction of TiB_2 .

3.6 Effect of reinforcement on wear behavior of the titanium-nickel based composite

Figure 7 shows the average coefficient of friction of composites at an applied load of 10 N. It is clearly shown that the addition of 5 vol.% Ni slightly decreases the coefficient of friction. Further addition of titanium diboride into the matrix exhibits a lower coefficient of friction compared to unreinforced commercial pure titanium. Reduction in the coefficient of friction upon addition of nickel and titanium diboride is because of lubricating action of TiB_2 on the surface of the titanium-based composite creating some layer on the rubbing surfaces which further diminishes slides taking place at the composite and ball contact surfaces [25]. The wear volume and wear rate of the titanium-based composites are presented in Fig. 7b-c. It is seen that the wear volume is responsive to the extent of the applied load. It is noticed that the wear volume decreases with increase in the

reinforcement (TiB_2) content, which can be attributed to the hardness of titanium-based composites which displayed an increase as the reinforcement contents are added into the titanium alloy.

Fig. 7. Graph showing the effect of reinforcement: (a) coefficient of friction, (b) wear volume and (c) wear rate

Conclusion

1. Homogenous dispersion of Ni-TiB₂ into the matrix alloy was accomplished. While the manufacture of titanium-nickel based composite from powder metallurgy utilizing spark plasma sintering method was consolidated.
2. Addition of alloying element and ceramic particles (Ni-TiB₂) to the titanium matrix significantly influence through increasing the microhardness value and causing a transformation in the microstructure by hindering grain development and grain boundary movement. However, there was a decrease in the density of the produced titanium matrix composite with an increase in the concentration of reinforcement.

3. The microstructural evolution shows the transformation from lamellar into an equiaxed phase with titanium diboride situated at the grain boundaries of the titanium matrix composite. There is additionally a good interfacial bonding between the titanium matrix and both Ni-TiB₂ support materials.
4. Titanium-nickel based composites exhibited good wear resistance when related to commercially pure titanium. Increase in the reinforcement addition displayed a lower coefficient of friction compared to titanium alloy.

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Figures

Figure 1

SEM morphology of the powders; (a) Commercial pure titanium, (b) Nickel, (c) Titanium diboride, (d-e) admixed powders of Ti-Ni with 5 and 20 vol.% TiB₂ and (f) EDX of the admixed powders.

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Figure 2

XRD patterns of the admixed composite powders

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Figure 3

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Figure 4

(a) Relative density and (b) Microhardness values of the sintered titanium-based composite.

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Figure 5

SEM microstructure of sintered composites; (a) Ti alloy, (b) Ti-5 vol.% Ni, (c) Ti-5 vol.% Ni-TiB₂, (d) Ti-10 vol.% TiB₂, (e) Ti-15 vol.% TiB₂ and (f) Ti-5 vol.% Ni-20 vol.% TiB₂.

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